# metal-organic papers

# Bis(2-methyl-2-propanammonium) chromate

**Hammouda Chebbi and Ahmed Driss***

Laboratoire de Matériaux et Cristallographie, Département de Chimie, Faculté des Sciences de Tunis, 2092 El Manar II, Tunis, Tunisia

Correspondence e-mail: ahmed.driss@fst.rnu.tn

**Key indicators**

Single-crystal X-ray study
$T = 293$ K
Mean $\sigma$(C–C) = 0.003 Å
Disorder in solvent or counterion
$R$ factor = 0.036
$wR$ factor = 0.090
Data-to-parameter ratio = 15.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

A new organic chromate, $[(CH_3)_3CNH_3]_2[CrO_4]$, associated with the monoprotonated 2-methyl-2-propanamine molecule, has been synthesized. The structure of the compound consists of discrete chromate ions stacked in layers perpendicular to the (010) plane, separated by organic layers containing $[(CH_3)_3CNH_3]^+$ groups. The cohesion and stability of the structure are ensured by a two-dimensional network of hydrogen bonds in the (001) plane, where the O atoms of the anion are acceptors from the 2-methyl-2-propan-ammonium N—H groups.

## Comment

The present description of the bis(2-methyl-2-propan-ammonium)chromate structure, (I), is part of an investigation of materials resulting from interaction between chromic acid and organic molecules, such as amines and aminoalcohols.



(I)
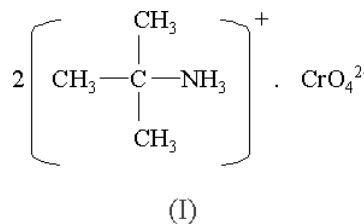
Among the investigated materials, we have previously described 2,2-dimethyl-1,3-propanediammonium chromate (Chebbi *et al.*, 2000), 4-ammonio-2,2,6,6-tetramethyl-piperidinium chromate dihydrate (Chebbi & Driss, 2001) and 1,4-butanediammonium chromate (Chebbi & Driss, 2002). Furthermore, the literature gives some examples of $CrO_4^{2-}$ associated with organic cations: $2(CN_3H_6)^+\cdot CrO_4^{2-}$ (Cygler *et al.*, 1976) and $2[(CH_3)_4N]^+\cdot CrO_4^{2-}\cdot xH_2O$ ($x = 0.5$ or 2; Sorehkin *et al.*, 1978). Two components, inorganic $CrO_4^{2-}$ and organic $[(CH_3)_3CNH_3]^+$, constitute the atomic arrangement of $[(CH_3)_3CNH_3]_2[CrO_4]$, (I) (Fig. 1). Six chromate layers per cell are parallel to (001) planes at $z_i = 2i + 1/12$ ($0 \le i \le 5$), while the organic groups provide the cohesion in layers through N—H···O hydrogen bonds (Fig. 2). The network of $CrO_4^{2-}$ lies between two networks of cations with no inter-connection between successive layers. The $CrO_4^{2-}$ anion has local $3m$ symmetry instead of regular $\overline{4}3m$ symmetry and the charge is compensated by the 2-methyl-2-propanammonium cation. The Cr atom is disordered over two positions, separated by 0.9 Å and each having an occupancy of 0.5. The Cr—O distances within the distorted $CrO_4$ tetrahedron vary from 1.620 (5) to 1.632 (2) Å. In addition, the values of the O—Cr—
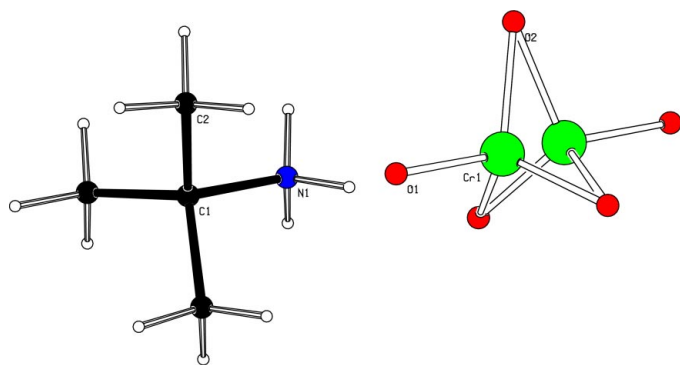
DOI: 10.1107/S1600536802012412

**Figure 1**
The molecular structure of (I) (*PLATON*; Spek, 1998). Atoms Cr1 and O1 have a site-occupation factor of 0.5.

O angles [106.77 (4)–112.03 (3)°] differ significantly from the ideal value of 109.5°. These values are generally observed for this type of tetrahedron (Gerault *et al.*, 1987; Bars *et al.*, 1977; Stephens & Cruickshank, 1970; de Brauer *et al.*, 1991). With regard to the geometry of the anion, there is a slight dissymmetry in the Cr—O bond lengths; the Cr—O1 bond is significantly shorter than the other three. This probably reflects the fact that atom O1 is not involved in any hydrogen bonding. The 2-methyl-2-propanammonium groups establish hydrogen bonds involving the H atoms of the $NH_3$ groups: one N—H⋯O bond has an N⋯O distance of 2.806 (2) Å and participates in the cohesion of the two-dimensional network. The N—C and C—C distances, and C—C—N and C—C—C angles in this organic group are comparable whith those observed for other compounds (Cygler *et al.*, 1976; Chebbi *et al.*, 2000; Chebbi & Driss, 2001, 2002).

## Experimental

The title compound was prepared from a 1:2:100 mixture of $CrO_3$ (2 g), $C_4H_{11}N$ (4.84 g) and $H_2O$ (36 g). Yellow single crystals, suitable of X-ray analysis, were obtained from the solution by slow evaporation of the solvent at room temperature.

### Crystal data

| | |
|---|---|
| $(C_4H_{12}N)_2[CrO_4]$ | Mo $K\alpha$ radiation |
| $M_r = 264.29$ | Cell parameters from 25 |
| Trigonal, $R\bar{3}c$ | reflections |
| $a = 6.720 (1)$ Å | $\theta = 11$–$14°$ |
| $c = 54.330 (9)$ Å | $\mu = 0.81$ mm$^{-1}$ |
| $V = 2124.8 (6)$ Å$^3$ | $T = 293 (2)$ K |
| $Z = 6$ | Hexagonal prism, yellow |
| $D_x = 1.239$ Mg m$^{-3}$ | $0.28 \times 0.25 \times 0.14$ mm |

### Data collection

| | |
|---|---|
| Enraf–Nonius CAD-4 | $R_{int} = 0.057$ |
| diffractometer | $\theta_{max} = 27.0°$ |
| $\omega/2\theta$ scans | $h = 0 \rightarrow 8$ |
| Absorption correction: $\psi$ scan | $k = -8 \rightarrow 0$ |
| (North *et al.*, 1968) | $l = -69 \rightarrow 69$ |
| $T_{min} = 0.802$, $T_{max} = 0.893$ | 2 standard reflections |
| 1936 measured reflections | frequency: 120 min |
| 524 independent reflections | intensity decay: 0.4% |
| 359 reflections with $I > 2\sigma(I)$ | |
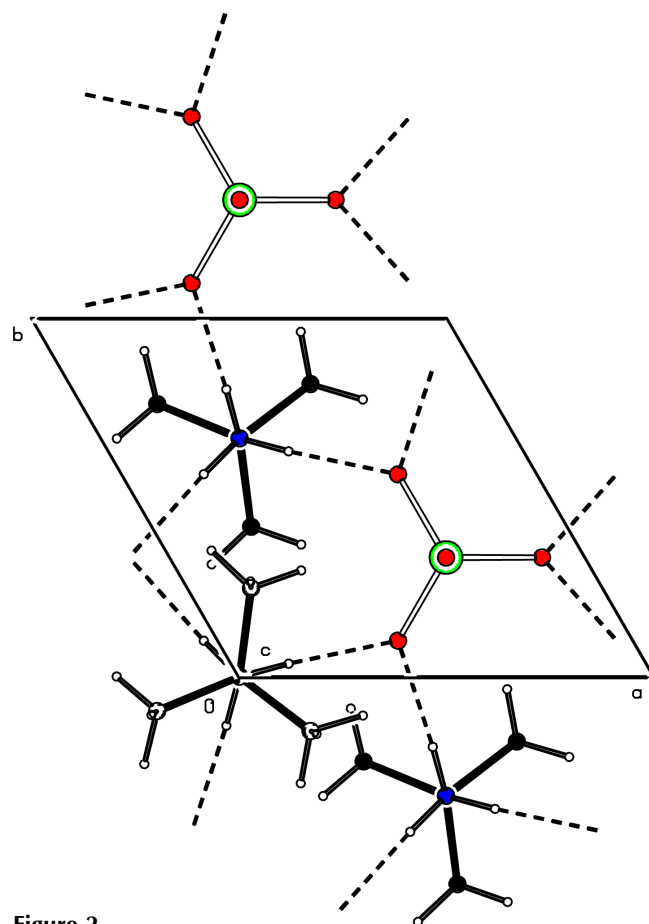


**Figure 2**
Projection of (I) along the *c* direction, with dashed lines indicating hydrogen bonds.

### Refinement

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.02P)^2$ |
| $R[F^2 > 2\sigma(F^2)] = 0.036$ | $+ 0.149P]$ |
| $wR(F^2) = 0.091$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.37$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 524 reflections | $\Delta\rho_{max} = 0.17$ e Å$^{-3}$ |
| 35 parameters | $\Delta\rho_{min} = -0.16$ e Å$^{-3}$ |
| H atoms treated by a mixture of | |
| independent and constrained | |
| refinement | |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Cr1—O1 | 1.620 (5) | N1—C1 | 1.505 (4) |
| Cr1—O2 | 1.632 (2) | C1—C2 | 1.524 (3) |
| | | | |
| O1—Cr1—O2 | 106.77 (4) | N1—C1—C2 | 107.17 (17) |
| O2$^i$—Cr1—O2 | 112.03 (3) | C2$^{ii}$—C1—C2 | 111.67 (15) |

Symmetry codes: (i) $1 - y, x - y, z$; (ii) $1 - y, 1 + x - y, z$.

**Table 2**
Hydrogen-bonding geometry (Å, °).

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| N1—H4⋯O2$^i$ | 0.92 (2) | 1.88 (2) | 2.806 (2) | 176 (2) |

Symmetry code: (i) $x, 1 + y, z$.

H atoms of methyl groups were placed at calculated positions and refined as riding, with C—H = 0.96 Å. Atom H4, bonded to N, was located in difference Fourier syntheses and was refined isotropically. The Cr atom is disordered over two positions, separated by about 0.9 Å, and these positions were refined with half occupancies. The site-occupancy factor of the chromate atom O1 was refined and found to be close to 0.5 and so was fixed at 0.5 for further refinement as there was a large correlation between the displacement parameters and the site-occupancy factor in the refinement calculations.

Data collection: *CAD*-4 *EXPRESS* (Duisenberg, 1992; Macíček & Yordanov, 1992); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1998).

## References

Bars, O., Le Marouille, J.-Y. & Grandjean, D. (1977). *Acta Cryst.* B**33**, 3751–3755.

Brauer, C. de, Jannin, M., Puget, R. & Perret, R. (1991). *Acta Cryst.* C**47**, 2231–2232.

Chebbi, H., Hajem, A. A. & Driss, A. (2000). *Acta Cryst.* C**56**, e333–e334.

Chebbi, H. & Driss, A. (2001). *Acta Cryst.* C**57**, 1369–1370.

Chebbi, H. & Driss, A. (2002). *Acta Cryst.* E**58**, m147–m149.

Cygler, M., Grabowski, M. J., Stepien, A. & Wajsman, E. (1976). *Acta Cryst.* B**32**, 2391–2395.

Duisenberg, A. J. M. (1992). *J. Appl. Cryst.* **25**, 92–96.

Fair, C. K. (1990). *MolEN*. Enraf–Nonius, Delft, The Netherlands.

Gerault, Y., Riou, A., Cudennec, Y. & Bonnin, A. (1987). *Rev. Chim. Miner.* **24**, 631–640.

Macíček, J. & Yordanov, A. (1992). *J. Appl. Cryst.* **25**, 73–80.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Sorehkin, V. N., Tabachenko, V. V. & Serezhkina, L. B. (1978). *Himia I Himiceskaa Technol.* **21**, 292–292.

Spek, A. L. (1998). *PLATON*. Utrecht University, The Netherlands.

Stephens, J. S. & Cruickshank, D. W. J. (1970). *Acta Cryst.* B**26**, 437–439.